



Calhoun: The NPS Institutional Archive
DSpace Repository

Theses and Dissertations

1. Thesis and Dissertation Collection, all items

1965

Deformation and imperfections in aluminum.

Randall, Robert H.

Monterey, California: U.S. Naval Postgraduate School

<http://hdl.handle.net/10945/11925>

This publication is a work of the U.S. Government as defined in Title 17, United States Code, Section 101. Copyright protection is not available for this work in the United States.

Downloaded from NPS Archive: Calhoun



<http://www.nps.edu/library>

Calhoun is the Naval Postgraduate School's public access digital repository for research materials and institutional publications created by the NPS community. Calhoun is named for Professor of Mathematics Guy K. Calhoun, NPS's first appointed -- and published -- scholarly author.

Dudley Knox Library / Naval Postgraduate School
411 Dyer Road / 1 University Circle
Monterey, California USA 93943

NPS ARCHIVE
1965
RANDALL, R.

DEFORMATION AND IMPERFECTIONS
IN ALUMINUM

ROBERT H. RANDALL

DEFORMATION AND IMPERFECTIONS
IN
ALUMINUM

* * * * *

Robert H. Randall

DEFORMATION AND IMPERFECTIONS

IN

ALUMINUM

by

Robert H. Randall

//

Lieutenant, United States Naval Reserve

Submitted in partial fulfillment of
the requirements for the degree of

MASTER OF SCIENCE

IN

MATERIALS SCIENCE

United States Naval Postgraduate School
Monterey, California

1 9 6 5

DEFORMATION AND IMPERFECTIONS
IN
ALUMINUM

by
Robert H. Randall

This work is accepted as fulfilling
the thesis requirements for the degree
MASTER OF SCIENCE
from the
United States Naval Postgraduate School

ABSTRACT

The occurrence of twinning in aluminum and the part played by twinning in the plastic deformation of aluminum has not been adequately interpreted. The structure of aluminum has been determined only by the Debye-Scherrer powder method which does not reveal the presence of twinning. With the use of oscillating crystal techniques, the twinning is revealed in aluminum. With the demonstration of twinning in aluminum, the need for further analysis of twinning as a mode of deformation becomes more and more apparent.

TABLE OF CONTENTS

Section	Title	Page
1.	Introduction	1
2.	Discussion	5
3.	Experimental Procedure	14
4.	Experimental Results	17
5.	Conclusions	27
6.	Acknowledgements	28
7.	Bibliography	29

LIST OF ILLUSTRATIONS

Figure		Page
1.	Stacking Sequences	7
2.	Translations leading to variations in stacking sequences	9
3.	Dislocation dissasociation	11
4.	Laue pattern of aluminum	18
5.	Laue pattern of aluminum	19
6.	Micro-photometer trace	20
7.	Oscillating crystal pattern	21
8.	Oscillating crystal pattern	22
9.	Oscillating crystal pattern	23
10.	Indexed oscillating crystal pattern	25

SECTION 1

INTRODUCTION

Metals in service may be and during fabrication are subjected to stresses tending to pull the metal apart, to cause one part to slide over another, to compress it, or to twist it. These stresses lead to some change of shape in the body. All metals whether polycrystalline or single crystals, will deform to some extent under stress. There are several ways in which a metal may deform under this applied stress. The principle modes of plastic response to stresses are slip and twinning.

The most common mode of deformation in metal crystals is generally assumed to be slip. Slip occurs by the apparent sliding of blocks of the crystal over one another on definite planes called slip planes. It is now accepted that lattice defects called 'dislocations' are present and that they can move under the action of stresses. The motion of the dislocation produces a relative displacement between two parts of the crystal. Additional dislocations sweeping through the same plane add to the displacement, the over-all effect being slip.

Another mode of deformation is the formation of crystallographic twins. This process is very different from slip, since the orientation of the crystal above and below the slip plane is the same after slip, whereas, in twinning it is altered. Slip movements occur in multiples of a single atomic spacing,

whereas, the twinning movement is a shear, proportional to the distance from the twinning plane.

Crystallographically, twinning is described in terms of symmetry operations, rotations, reflections and inversions, but physically, twinning can be explained in terms of a mistake in the normal stacking sequence of a set of parallel planes in the crystal, creating a 'stacking fault'. If the fault ends inside the crystal, its boundary is a partial dislocation loop.

The problem discussed here is the occurrence of twins in aluminum and the part played by both slip and twinning in the plastic deformation of aluminum. Twins are infrequently observed in aluminum¹ which is unusual among the close-packed metals, i.e., silver, copper, cadmium, zinc, nickel, etc. This has been explained by postulating a high stacking fault energy for aluminum but stacking fault energies are not accurately measured or predicted. That this is the case has been demonstrated in this laboratory by the discovery that twinning is common, but unrecognizable, in alpha iron².

Twins are not generally observed in aluminum either by macroscopic or microscopic observation. The reason for not observing twins in aluminum could be twofold: (1) aluminum oxidizes fairly rapidly so that a layer of aluminum oxide would mask observation of twins; (2) if twins are present they may be extremely small so that even a careful examination would not reveal twinning³. If these small twins do exist, ordinary diffraction techniques have not revealed them. The fact that twinning is not immediately

observed by ordinary optical examination in aluminum is not conclusive evidence for a complete absence of twinning.

A search of the literature⁴⁻¹³ on crystal structure determination shows that single crystal methods have not been employed in determining the structure of aluminum. The reported structure determinations and precision lattice constant measurements for aluminum have been conducted by the De-Bye-Scherrer powder method. The DeBye-Scherrer powder method does not reveal the presence of twinning since diffraction from twinned crystals has not been analyzed in sufficient detail to differentiate scattering from twinned and un-twinned crystals.

The lack of observed twinning in aluminum has been said to be caused by high stacking fault energy. High stacking fault energies lead to low stacking fault probability. The postulation of an unexplained or unexpected high stacking fault energy is not an adequate explanation for the lack of twinning in aluminum. Twins and stacking faults are known to occur in the other close-packed metals whether they be cubic or hexagonal. There is no crystallographic reason why aluminum, a close-packed metal, should not twin readily and if there is a physical reason it is sufficiently complicated or obscure to have escaped detection to date.

The diffraction patterns of deformed aluminum, like all malleable crystals, show a marked blurring and elongation of the Laue spots known as 'asterism'. For many years plastic deformation was thought to be the

the cause of asterism. However, plastic deformation without asterism was observed by Rom and Kochendorfer¹⁴ in aluminum and by others in various other metals. It has been suggested that asterism is connected with kinking or deformation bands. Many workers have concluded that asterism is caused by reoriented regions; whether these reoriented regions are kinks, deformation bands, areas of small rotations, or twins has not been clearly understood or explained.

In spite of the development of dislocation theory in recent years, it cannot be said that the processes of plastic deformation, strain hardening and fracture are well understood. If aluminum differs from other ductile metals in that it does not twin readily it can be contrastingly compared with the others in which twinning is extensive. If aluminum, like iron, does twin readily, although not visibly, twinning as a mode of deformation requires more analysis than it has been given to date.

SECTION 2

DISCUSSION

Two crystals are said to have a twinned orientation with respect to each other, if given one, the other is related to it by a crystallographic symmetry operation which is not a symmetry element of the crystal lattice. This symmetry element may be a rotation about a given axis, or a reflection across a common plane, or an inversion through a center. The inversion through a center produces a new unit cell which is a simple multiple in size of the basic unit cell.

The two (or more) elements of this twinned edifice can be pictured as being stacked one upon another with an axis, plane or center common to both. Another way to picture this edifice is to create a synthetic lattice that will contain the two elements (or all possible twin orientations). There is a cell of this edifice, which, since it is a cell period of the homogeneous crystal, should be one of its simple multiples. The cell of this edifice would have elements of symmetry or of pseudosymmetry which do not belong to the individual components of this edifice. These elements of symmetry or pseudosymmetry are either of a higher or lower order than those of the individual elements of the edifice.

One gives the name mimetic structures to these complex edifices composed of twins, the exterior form obtained simulating more or less

exactly a symmetry higher or lower than that of the simple crystals composing it.³ In electron diffraction studies of thin layers of aluminum vapor deposited on platinum, Finch and Quarrell¹⁵ reported that they obtained aluminum exhibiting a face-centered tetragonal structure, with the axial ratio being 1.03. Yet they report that a sufficiently thick layer of aluminum gave the usual face-centered cubic structure. This could be the result of mimetic twinning. Imitative twinning of this kind is very frequent in orthorombic pseudo-hexagonal substances. H. A. Liebovitch² has shown that mimetic twinning exists in alpha iron. The basic structure of alpha iron being tetragonal, but due to mimetic twinning the overall appearance is cubic.

Twinned crystals have been observed in most close-packed metals, either in the deformed or annealed condition, or both. It is common to refer to the twins as deformation or mechanical twins and annealing twins respectively. The observation of annealing twins in metals has been extensively reviewed by Carpenter and Tamura¹⁶ and by Mathewson¹⁷.

The boundary between twinned crystals frequently is the twinning plane. Since neighboring atoms can match up across a twin boundary corresponding to the (111) plane in the face-centered cubic crystals, a boundary of this type is called a coherent twin boundary. Twin boundaries that do not coincide with the twin plane do not permit matching up of neighboring atoms and are therefore referred to as non-coherent twin boundaries.

A face-centered cubic crystal, considered as a layered structure produced by the stacking of close-packed planes, (111) planes, can contain three different types of stacking errors as shown by Read¹⁸.

1. Intrinsic fault
2. Extrinsic fault
3. Twin (growth) fault

The stacking sequence of these faults are shown below in Figure 1.

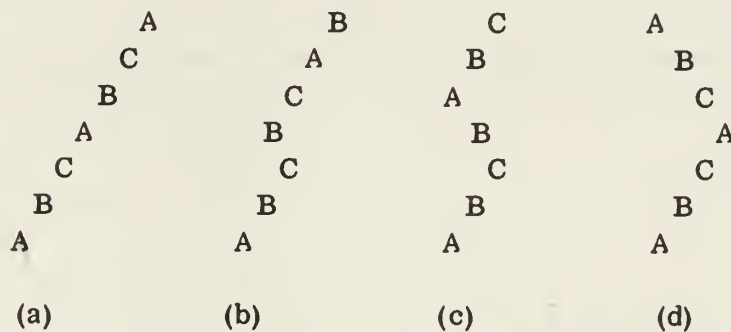


Figure 1. Stacking sequences for: (a) perfect crystal; (b) Intrinsic fault; (c) Extrinsic fault; (d) Twin fault.

The X-ray diffraction effects in polycrystalline materials of intrinsic faulting, twin faulting or a mixture of the two types have been studied extensively by Warren¹⁹ and students and others. An analysis of diffraction

effects of extrinsic faults in face-centered cubic crystals has been done by Johnson²⁰.

The layered structure in face-centered cubic crystals can be described by noticing that successive close-packed planes are offset by a constant vector. In the stacking of (111) planes there are two offset vectors that lead to close-packed structures: $\pm f = \pm (a_0/6) [\bar{1}\bar{2}1]$.

A stacking fault can be described as a variation in the normal sequence of offset factors. Stacking faults in face-centered cubic structures can be thought of as twins of one or more atomic layers thick. In a face-centered cubic structure this variation in the normal stacking sequence can be accomplished by three different translations on the (111) planes:

$$f_1 = \pm 1/6 [\bar{2}11]$$

$$f_2 = \pm 1/6 [1\bar{2}1]$$

$$f_3 = \pm 1/6 [11\bar{2}]$$

this is shown in Figure 2. These three basic translations operating on all eight (111) planes lead to a total of twenty-four possible twin orientations plus the original.

A stacking fault may not extend over the entire layer of the close-packed plane in a crystal. In such cases it must end at a type of dislocation that involves a displacement vector that is not a lattice vector; a dislocation of this type is either a Frank or a Shockley partial dislocation. When two partial

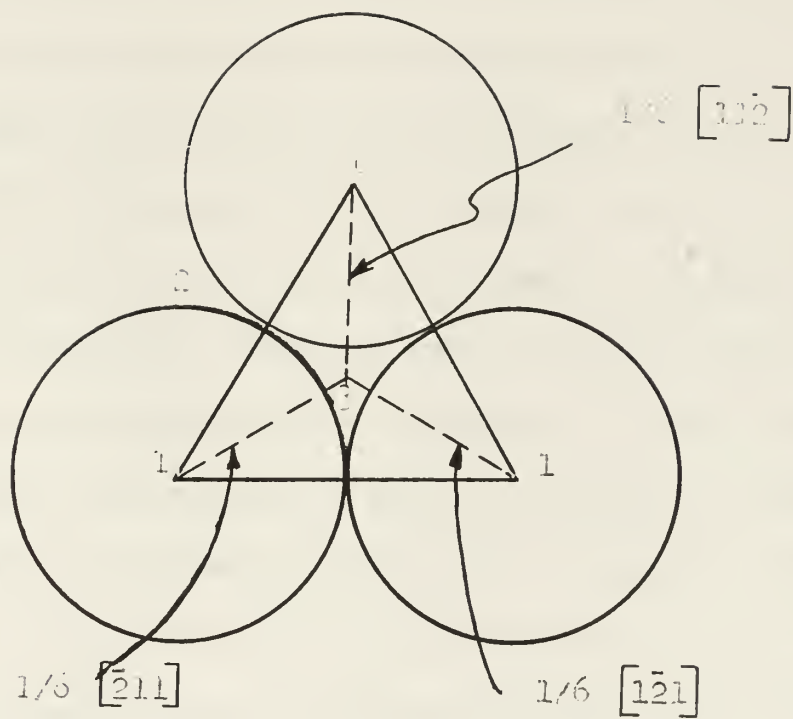


Figure 2. Face Centred Cubic (111) plane

dislocations of opposite sign move apart on the same slip plane they leave behind a stacking fault.

The energy of a stacking fault may be estimated in several ways:

(1) The energy of the stacking fault is proportional to the area between the two partial dislocations; (2) The stacking fault energy is proportional to the square of the Burgers vector describing the partial dislocation; (3) The stacking fault energy can be estimated by counting next-nearest neighbor violations (assuming next-next-nearest neighbor interactions to be unimportant energy wise); (4) The stacking fault energy has been estimated from the ratio of twin boundary energy to average grain boundary energy. Fullman²¹ has estimated the stacking fault energy for aluminum to be from 100 to 200 ergs/cm². All values given for stacking fault energy in aluminum are only approximations or estimates not accurate or reliable energies. The energies are said to be high on the basis of a lack of observed stacking faults (and therefore twins) in aluminum. The stacking fault energy in alpha iron is also said to be high for the same reason, but as previously mentioned twins (and therefore stacking faults) have been observed in iron in this laboratory.

Conditions that should be favorable to faulting are: (1) a low energy interface at the fault; and (2) a decrease in free energy in converting a section of the crystal from its original structure to the structure at the fault. The low interface requirement is met to some extent in aluminum

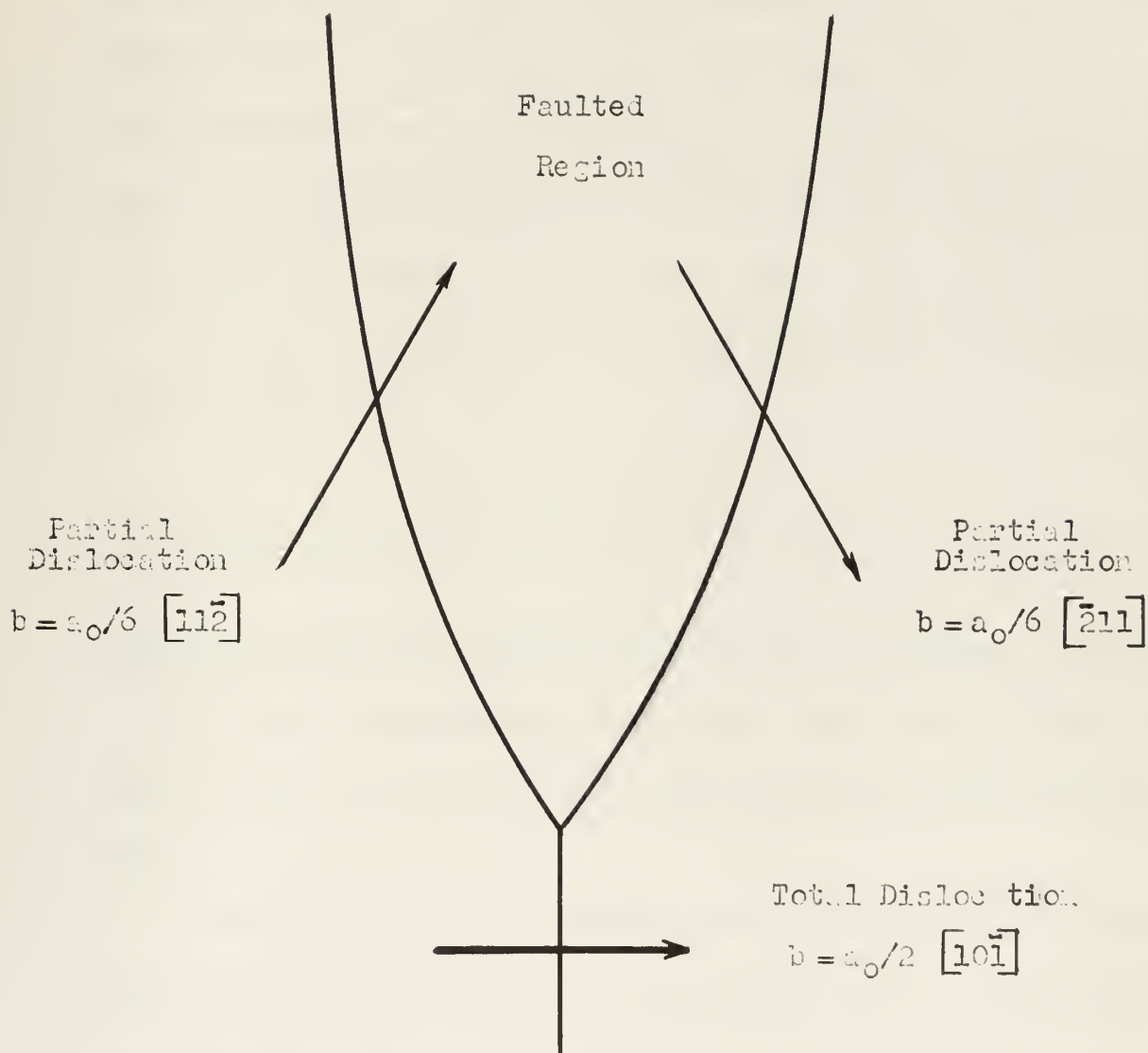


Figure 3. Dissociation of a total dislocation into two partial dislocations that have separated leaving a faulted region behind.

since a fault produces a twin on a (111) plane and since coherent twin boundaries are known to be low energy interfaces²².

In studying the structure of metals, the X-ray diffraction method is of primary importance. Any periodically arranged atomic structure will diffract X-rays. The diffraction pattern can be used to determine the specimens orientation and also its symmetry. The intensity of the diffracted beam is proportional to the square of the absolute value of the structure factor, $|F|^2$, wavelength, diffraction angle, etc., being constant.

The structure factor, 'F', for a set of planes (h,k,l) for a crystal is defined by equation 1.

$$F_{hkl} = \sum_{j=1}^n f_j e^{2\pi i (hx + ky + lz)} \quad (1)$$

where f is the electronic scattering factor for the j^{th} atom with coordinates (x, y, z,) the summation extending over the n atoms of the unit cell. The reader is referred to Cullity²³ for a complete discussion of the structure factor.

For the face-centered cubic cell, assuming it to contain four atoms of the same kind, the structure factor will be four if the indices (h,k,l,) are unmixed or if the indices are mixed the structure factor will be zero.

As previously discussed, based on the views of Freidel¹⁵, if there exists a twinned structure, the twinned structure should exhibit a cell that

is some simple multiple of the basic unit cell. This multiple unit cell in aluminum is three times the size of the basic unit cell. This means that instead of having one unit cell containing four atoms we now have twenty-seven unit cells containing four atoms for a total of one hundred and eight atoms in the basic structure. Each twinning orientation present is brought about by a shear as previously described on one of the (111) planes. For each twin orientation present we now have an additional one hundred and eight atoms in the structure. The problem now arises of mechanically summing over all the atoms present to determine the structure factor. This is a tremendous task and has been programmed for a computer. Preliminary results from the computer program indicate that the structure factor is not always zero or four, indicating that a difference in intensities of the diffracted beams is to be expected from a twinned structure.

SECTION 3

EXPERIMENTAL PROCEDURE

1. Specimen preparation

Single crystals of high purity aluminum were grown by the strain-anneal method. The crystal size obtained was $5/8" \times 1/2" \times 1/16"$. The crystal was cut out of the sheet and was mounted on a goniometer head in the standard fashion.

2. Experimental procedure

Using copper radiation (50 kilovolts and 40 milliamperes) filtered with a nickel filter, a series of Laue transmission patterns were taken traversing horizontally across the single crystal in approximately three millimeter steps. Each position was exposed for thirty minutes. Examining the diffraction patterns it can be seen that the relative intensities of certain spots change as we progress across the crystal. Measurement with a micro-photometer indicated that the same set of spots in patterns from different parts of the 'crystal' showed that the relative intensity of these spots differed by as much as a factor of two.

The 'crystal' was oriented with the apparent $[001]$ vertical and the (100) perpendicular to the X-ray beam, using the Laue transmission method and molybdenum radiation (50 kilovolts and 20 milliamperes) with zirconium filter.

After orientating the 'crystal', oscillating crystal patterns were taken. This method was chosen in order to avoid overlapping reflections, that are seen in rotating patterns, which lead to uncertainties in indexing and in determining the intensities of reflections. This method reduces the number of reciprocal lattice points that come into contact with the reflection sphere and so reduces the number of reflections. The 'crystal', oriented as described above, was oscillated through a fifty degree angle around this position. This method is described in standard texts such as Barrett²⁴ or Cullity²³.

Horizontal layer lines corresponding to the lattice parameter of aluminum, 4.04 angstroms, were measured. The diffraction patterns also showed intermediate layer lines. These intermediate layer lines were made up of spots and streaks. By changing the orientation slightly the same basic diffraction pattern was obtained but it was noted that a small change in orientation caused the appearance and disappearance of certain spots on the pattern.

To investigate the cause of the streaks noted on the diffraction patterns taken with molybdenum radiation, more diffraction patterns were taken with copper radiation (35 kilovolts and 20 milliamperes). First a pattern filtered with nickel to eliminate the beta radiation, and then one with an additional iron filter to preferentially absorb the longer wavelengths. The effect of the copper radiation was twofold: (1) copper characteristic radiation being of a longer wavelength than the molybdenum radiation

expanded the diffraction pattern enabling a better examination of the intermediate layer lines , and; (2) using the nickel filter and then a combination of iron and nickel filters showed that the diffracted spots and streaks were being caused by the K_{α} characteristic wavelengths and not by white radiation . The iron filter absorbed the longer wavelengths up to its absorption edge reducing the intensity of the streaks on the long wavelength side , whereas , the nickel filter eliminated the beta wavelengths . Neither filter absorbed the short wavelength white radiation apparently .

SECTION 4

EXPERIMENTAL RESULTS

1. Diffraction patterns taken scanning across the crystal

Figures 4 and 5 are two of the diffraction patterns taken by traversing across the aluminum single 'crystal'. Of interest here, as previously mentioned, is that the relative intensities of certain spots change as we traverse the 'crystal'. Figure 6 shows the micro-photometer examination of two sets of these spots. The difference in intensities can be explained in terms of a twinned structure. As we traverse across the 'crystal' the X-ray beam illuminates various twin orientations in the 'crystal'. As different combinations of twin orientations are illuminated, different combinations of planes of atoms will satisfy the diffracting conditions and differences in relative intensities will result.

2. Diffraction patterns taken by the oscillating crystal method

With the aluminum 'crystal' in a (001) orientation and with the $[001]$ direction in the vertical plane of the X-ray beam and perpendicular to it, a series of oscillating crystal patterns were taken. Figures 7, 8, and 9 are three such photographs.

It can be seen that with a small change in orientation the same basic diffraction pattern was obtained. But with this small change in orientation certain spots are seen to appear and disappear.



Figure 4. Laue Photograph
Specimen: Aluminum
Radiation: Cu(50kv, 40ma)
Nickel filter



Figure 5. Laue Photograph
Specimen: Aluminum
Radiation: Cu(50kv, 40ma)
Nickel filter

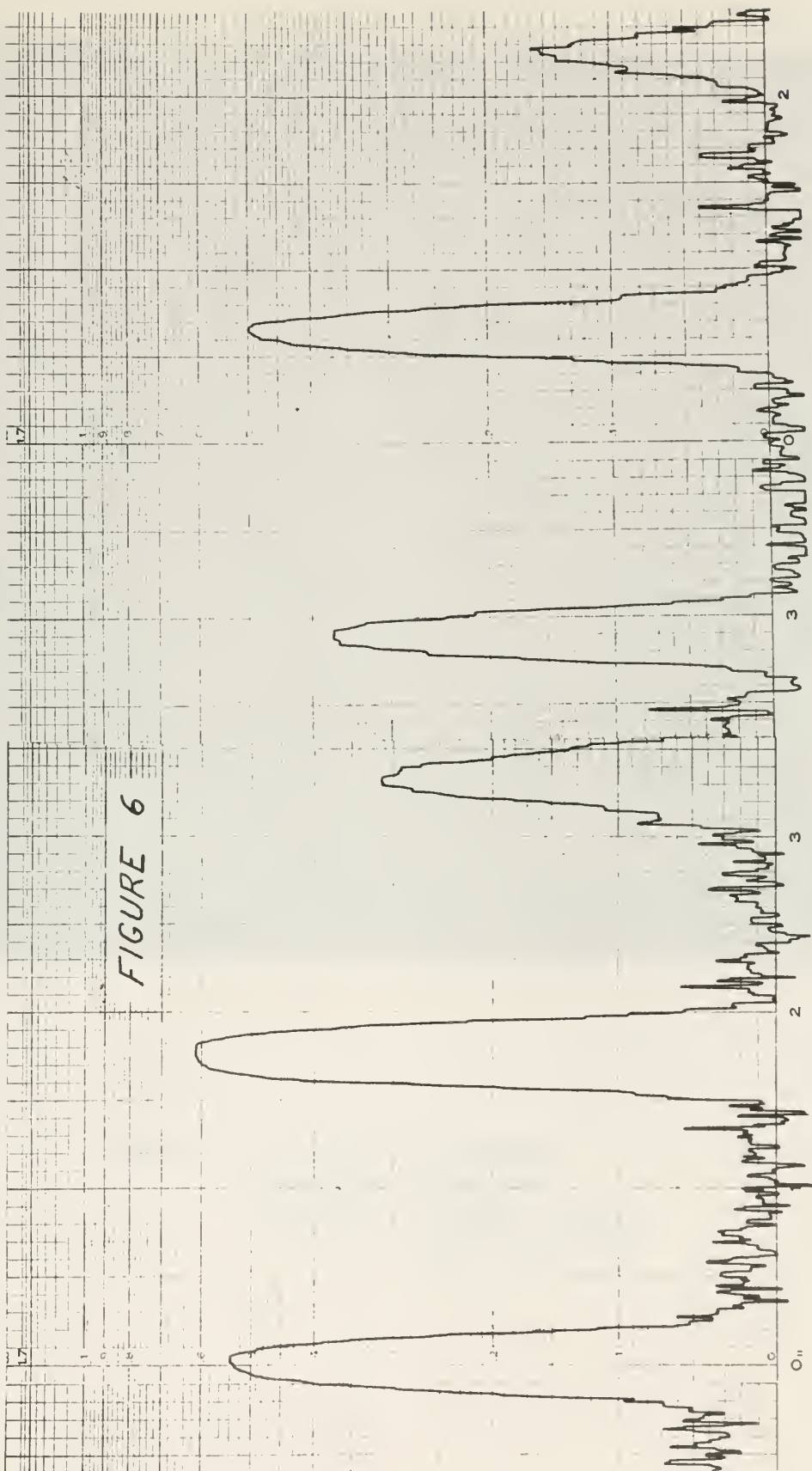




Figure 7. Oscillation Photograph
Specimen: Aluminum
Radiation: Mo(35kv, 20ma)
Zirconium filter



Figure 8. Oscillation Photograph
Specimen: Aluminum
Radiation: Mo(35kv, 20ma)
Zirconium filter



Figure 9. Oscillation Photograph
Specimen: Aluminum
Radiation: Mo(35kv, 40ma)

This is due to the reflection circle intersecting a lattice node as the 'crystal' is tilted sufficiently from its original position.

The diffraction patterns taken with molybdenum radiation were indexed using radial and Bernal charts. The Bernal chart was used to relate the spots on the diffraction pattern to points in the reciprocal lattice. A discussion of reciprocal space and the interpretation of oscillating crystal photographs is given in Barrett²⁴ and similar texts.

The reciprocal lattice is cut by the reflection sphere as it oscillates about the axis of rotation. The sphere swings through the angle of oscillation and sweeps out two cup-shaped regions in the reciprocal lattice. All points lying in these cup-shaped regions correspond to planes that are recorded in the oscillating diffraction pattern and can be readily indexed.

Certain spots, the (400), for example, lie outside the area swept out by the reflection sphere, yet, on indexing the diffraction pattern a streak appears at the (400) position. If the pattern is indexed in terms of a triple sized unit cell (Figure 10 is a blown up version of Figure 7, indexed in terms of this triple sized unit cell), where the interlayer distance is one-eighteenth of the side of the triple unit cell, all the points and streaks can be indexed. The spots that show on the diffraction pattern correspond to the structure of an untwinned single crystal, whereas, the streaks result from the intersection of the reflection sphere with the reciprocal lattice points of the twinned structure which may be rods in reciprocal space associated

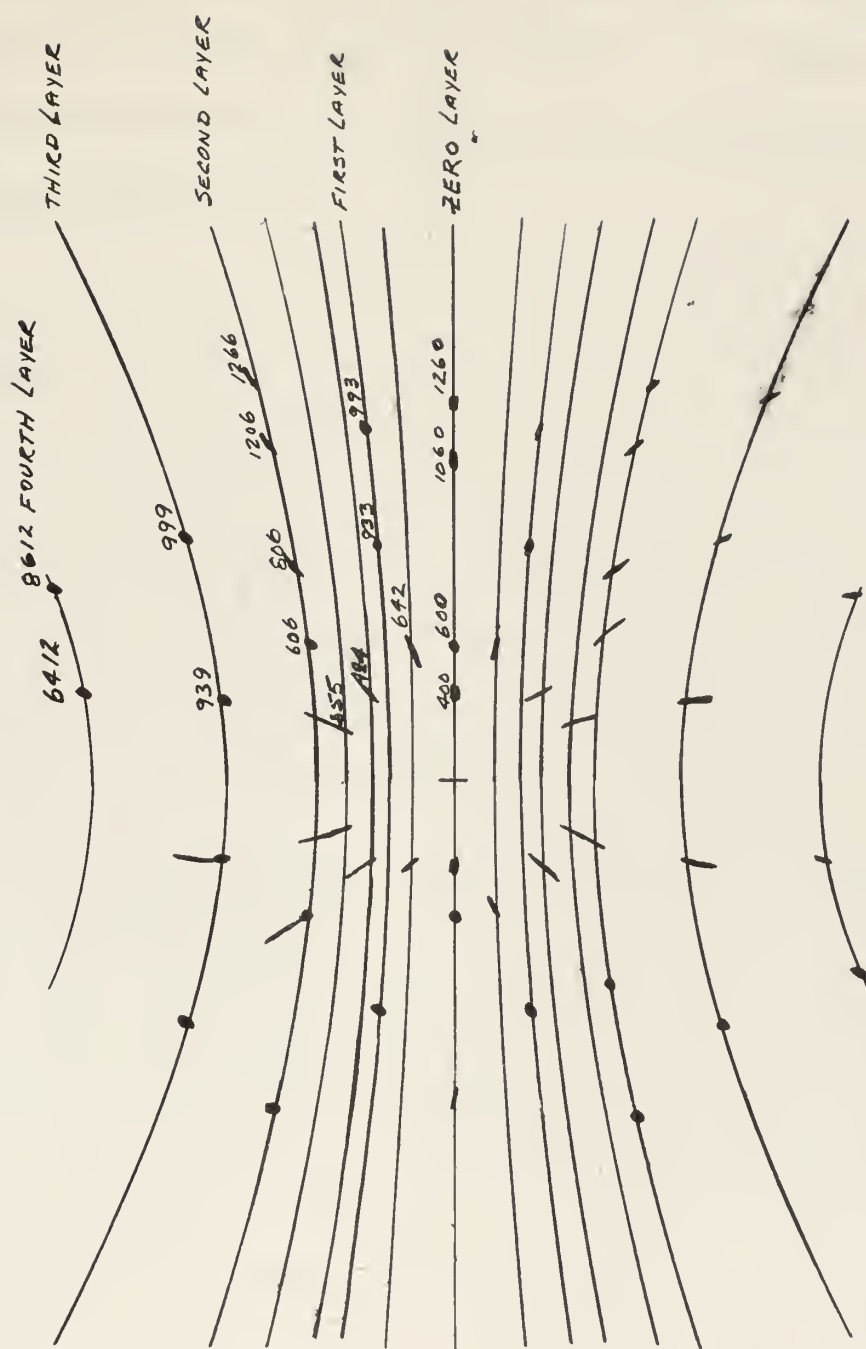


Figure 10. Oscillation Pattern

Fig. 7 indexed in terms of triple sized unit cell. Labelled layer lines are those of the untwinned single crystal.

with stacking faults on the $\{111\}$ planes. When the final results of the aforementioned computer program are complete we should be able to predict on the basis of the structure factor which twin orientations are present and which are not.

SECTION 5

CONCLUSIONS

Sufficient evidence for the presence of twinning in aluminum has been shown. The twinning, although not optically visible, can be demonstrated by the use of the oscillating crystal technique. Since aluminum is a malleable metal, and now that twinning in aluminum has been demonstrated, the statement that 'slip is the common mode of deformation' needs to be reevaluated in terms of this new evidence.

It is believed that further work needs to be done in the accurate determination of the structure of many metals, as the powder technique, unless properly evaluate, does not reveal the detailed structure of the metal.

Also further work needs to be done in the area of controlling twins, and twin size. With the discovery of the presence of twinning in aluminum it appears that twinning plays a much more important role in the deformation process than has been heretofore thought.

SECTION 6

ACKNOWLEDGEMENTS

My advisor, J. R. Clark, without whom this investigation would not have been done, for his continuing helpful advice and encouragement.

Mr. Roy Edwards for his very helpful assistance during the investigation.

SECTION 7

Bibliography

1. Cahn, R. W., *Advances in Physics*, v.3, no. 12, 1954.
2. Liebovitch, H. A., *Masters Thesis*, U. S. Naval Postgraduate School, 1965.
3. Friedel, G., *Lecons de Cristallographie*, Paris: Berger-Levrault, 1926.
4. Davey, W. P., *Phys. Rev.*, 25, 753, 1925.
5. Owen, E. A. and E. L. Yates, *Phil. Mag.*, 21, 809, 1936.
6. Jette, E. R. and F. Foote, *J. Chem. Phys.*, 3, 605, 1935.
7. Jevins, A. and M. Straumanis, *Z. Phys. Chem.*, B33, 265, 1936; B34, 402, 1936.
8. Owen, E. A., *Structure Reports*, V111, 3, 1948.
9. Kochanovska, *Structure Report*, X1, 4, 1947.
10. Donnay, J. D. H., G. Donnay, E. G. Cox, O. Kennard, M. V. King, *Crystal Data Determinative Tables*, American Crystallographic Association, 1963.
11. Wyckoff, R. W. G., *Crystal Structures*, Interscience, 1948.
12. Dana, E. S., *Textbook of Mineralogy*, Wiley, 1932.
13. *Strukturbericht*, v.1, 43; v.2, 171; v.3, 182, 187, 201; v.4, 82.
14. Rom, F. and A. Kochendorfer, *Z. Metallk.*, 41, 265, 1950.
15. Finch, G. I. and A. G. Quarrell, *Proc. Roy. Soc.*, A, f. 141, 398, 1933.

16. Carpenter, H. C. H. and S. Tamura, Proc. Roy. Soc., A, 113, 161, 1926.
17. Mathewson, C. H., Proc. Inst. Metals Div., Am. Inst. Mining and Met. Engrs., 7, 1928, 1928.
18. Read, W. T., Dislocations in Crystals, New York, McGraw-Hill, 1953.
19. Warren, B. E., Prog. Met. Phys., 8, 147, 1959.
20. Johnson, C. A., Acta. Cryst., 16, 490, 1963.
21. Fullman, R. L., J. Applied Phys., 22, 448, 1951.
22. Barrett, C. S., G. Ansel, R. F. Mehl, Trans. Am. Soc. Metals, 25, 702, 1937.
23. Cullity, B. D., Elements of X-Ray Diffraction, Addison-Wesley, 1959.
24. Barrett, C. S., Structure of Metals, McGraw-Hill, 1943.
25. Guinier, A., X-Ray Diffraction, Freeman, 1963.

thesR205

Deformation and imperfections in aluminu



3 2768 002 05277 1

DUDLEY KNOX LIBRARY